On Demand Analysis: Glycols by LC/MS/MS
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Region III's HPLC/MS/MS method for glycols is based on modifications to ASTM D D7731 – 11, and EPA Methods SW-846-8000C and SW-846- 8321. The method is under development with the intent to eventually have a validated, documented method. Aqueous samples are injected directly on the HPLC after tuning the MS/MS with authentic standards. This method was developed to detect and quantitate 2-methoxyethanol, 2-butoxyethanol, diethylene glycol, triethylene glycol, and tetraethylene glycol in aqueous samples.

The HPLC/MS/MS system is a Waters TQD. The HPLC column used to separate the analytes is a Waters (Milford MA) Atlantis dC18 3um, 2.1 x 150mm column (p/n 186001299). The HPLC gradient is with H20 and CH3CN with 0.1% formic acid. All details of instrument conditions are included in the case file. A custom standard mix from Ultra Scientific, (Kingstown Rl) is used for the instrument calibration. The working, linear initial calibration range varies for each compound, but is generally between 10-500 ppb and may change with further development. Reporting limits are between 10 and 50 ppb. Initial calibration (IC) is performed before each day's sample set; calibration verification is done at the beginning, after every 10 sample injections, and at the end of a sample set. The system is tuned with individual authentic standards of each compound according to the manufacturer's directions using the Waters Empower "Intellistart" tune/method development program in the MRM (multiple reaction monitoring) ESI+ (electrospray positive) mode. A custom standard mix supplied by Accustandard (New Haven, CT), is used as a second source verification(SSV) for the initial calibration. The SSV is run after IC. Other quality control measures include method blanks, field blanks, lab fortified blanks and matrix spikes and matrix spike duplicates.

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